CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

[redacted]

Mines Branch

COPY NO. 8

CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

MINES BRANCH INVESTIGATION REPORT

IR 74-32 July, 1974

SUITABILITY OF A QUEBEC GROUP SHALE FOR VITRIFIED STRUCTURAL CLAY PRODUCTS

by

K.E. Bell

Mineral Processing Division

Mines Branch Investigation Report IR 74-32

SUITABILITY OF A QUEBEC GROUP SHALE FOR VITRIFIED STRUCTURAL CLAY PRODUCTS

by

K.E. Bell*

- - -

SUMMARY OF RESULTS

National Sewer Pipe Limited, through their subsidiary, Montreal Terra Cotta Limited, proposes to develop a deposit of shale near St-Apollinaire, Que., for use in the manufacture of flue liners and vitrified clay sewer pipe at their Deschaillons plant.

Four samples from a surface exposure were selected for examination, based on their distinctive appearance. Two of the samples, a red and a red-and-black shale, showed excellent fired properties and firing behaviours, but minimally acceptable plasticity and low dry strengths. A sample of hard black shale had unsatisfactory plastic properties and was too open-firing for vitrified products, probably owing to a high coal content: it appears to be a very minor component of the deposit and its adverse effects should be insignificant due to dilution. A grey shale adjacent to the main deposit showed unsatisfactory plastic and firing behaviours, and it is recommended that this material be avoided through selective mining procedures.

Additions of plastic clay from the existing pit at the Deschaillons plant markedly improved the plastic behaviour and increased the dry strength of the red shale, which is the major component of the deposit. The optimum mixture probably contains 20 to 25 per cent clay, which showed good extrusion and safe drying behaviours. Dry moduli of rupture were considered to be adequate for safe handling of large ware such as sewer pipe and flue liners. Although the safe firing range for suitably dense ware was slightly narrowed, it remained in excess of 100 Fahrenheit degrees, more than sufficient for good process control.

---

*Research Scientist, Ceramic Section, Mineral Processing Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, Canada.

To evaluate the uniformity of the deposit, two 50-foot deep holes were core-drilled near opposite ends of the proposed pit site. In the absence of obvious natural discontinuities in the cores, they were divided for testing at 10-foot incremental depths. Although DTA indicated minor differences in clay mineral content between the holes, there was no apparent effect on refractoriness or on the fired properties of extruded specimens, which were remarkably similar. Within each hole, the DTA curves indicated little change with depth. However, with increasing depth, increased hardness and reduced plasticity were noted. The latter differences are unlikely to be significant in mixtures with plastic clay. Again, variations in refractoriness and fired properties with depth were noteably small.

Direction des mines

Rapport de recherches IR 74-32

ETUDE POUR VERIFIER SI UN GROUPE DE SCHISTES ARGILEUX PEUT CONVENIR A LA FABRICATION DE PRODUITS D'ARGILE VITRIFIEE

par

K.E. Bell*

RESUME DES RESULTATS

La National Sewer Pipe Limited, par l'intermédiaire de sa filiale, la Montréal Terra Cotta Limited, a l'intention d'exploiter un dépôt de schistes argileux situé près de St-Apollinaire (Québec) pour fabriquer des conduits de cheminée et des tuyaux d'égout en argile vitrifiée à leur usine de Deschaillons.

Sur un affleurement, on a choisi quatre échantillons d'apparence différente afin de les étudier. Deux de ces échantillons, l'un de schiste argileux rouge et l'autre rouge et noir, ont très bien réagit à la cuisson et possèdent d'excellentes propriétés une fois cuits, cependant ils ont fait preuve d'un minimum de plasticité, et de résistances à sec peu élevées. Un échantillon de schiste argileux noir et dur n'a pas démontré de propriétés plastiques satisfaisantes et sa réaction est trop mauvaise en contact direct avec la flamme pour permettre la fabrication de produits vitrifiés ce qui est probablement dû à une haute teneur en charbon: ce genre de schiste argileux ne représente qu'une infime partie du dépôt et la dilution devrait rendre ses effets négatifs insignifiants. Un schiste argileux gris, adjacent au dépôt principal a montré des propriétés plastiques insatisfaisantes et une réaction à la cuisson également insatisfaisante; il est recommandé d'éviter ce matériau en utilisant des méthodes sélectives d'exploitation.

Des additions d'argile plastique provenant de l'exploitation existante de Deschaillons ont amélioré d'un façon marquée les propriétés plastiques et la résistance à sec du schiste argileux rouge qui constitue la principale partie du dépôt. Le mélange optimal qui a fait preuve d'un bon extrudage et d'une réaction normale au séchage, contient probablement de 20 à 25 pour cent d'argile. Les coefficients de rupture sont considérés suffisants

---

*Chercheur Scientifique, Section de la Céramique, Division du Traitement des Minéraux, Direction des Mines, Ministère de l'Energie, des Mines et des Ressources, Ottawa, Canada.

pour permettre la manipulation sans danger de produits volumineux comme les conduits de cheminée et les tuyaux d'égout en argile vitrifiée. Même si la gamme de cuisson des produits suffisamment denses a dû être légèrement rétrécie, elle demeure de plus de 100°F, ce qui est plus que suffisant pour permettre un contrôle adéquat du procédé.

Pour évaluer l'uniformité du dépôt, on a prélevé des carottes dans deux trous de 50 pieds de profondeur près des extrimités opposées de l'emplacement proposé de l'exploitation. Comme les carottes n'ont pas fait voir de discontinuités naturelles évidentes, elles ont été divisées à tous les 10 pieds d'accroissement de la profondeur afin de leur faire subir des essais. Même si le DTA a indiqué des différences mineures dans la teneur en minéraux de l'argile des différents trous, elles n'ont pas eu d'effets apparents sur la nature réfractaire ou sur les propriétés après cuisson des échantillons soumis à l'extrusion, qui étaient remarquablement semblables. Pour chacun des trous, les courbes du DTA ont indiqué peu de changement suivant la profondeur. Cependant, à mesure que la profondeur augmentait on a remarqué une augmentation de la dureté et une diminution de la plasticité. Ces dernières différences seront probablement sans importance dans les mélanges avec l'argile plastique. Il faut répéter que le changement dans la nature réfractaire et les propriétés après cuisson des échantillons varient remarquablement peu selon la profondeur.

CONTENTS

INTRODUCTION

In the spring of 1973, National Sewer Pipe Limited, Oakville, Ontario, purchased Montreal Terra Cotta (1966) Ltd., operating a brick and tile plant at Deschaillons, Quebec. Substantial renovation of the existing manufacturing facility is planned, along with the introduction of the manufacture of vitrified clay sewer pipe, for which purpose it is proposed to develop a deposit of shale near St-Apollinaire, Quebec. In addition, the company hopes to market some dried, pulverized shale for use in the manufacture of pottery of the earthenware type. Surface samples of this shale had been examined in 1965* at the request of National Sewer Pipe, and had been reported on in considerable detail in 1966**. Evaluation of these samples indicated that the material should be suitable for the manufacture of vitrified clay pipe. To assure the availability of a sufficient supply of uniform material to merit developing the property, the company proposed to conduct a core-drilling program and requested assistance from the Mineral Processing Division in interpreting and evaluating the cores.

In November 1973, the writer visited the site of the proposed pit, in the company of Mr. H.G. Peets of National Sewer Pipe Limited and Mr. R. Corriveau of Montreal Terra Cotta Limited. Two properties, about ¼ mile apart, were examined, both at the eastern extremity of the village of St-Apollinaire.

---

*Test Report MPT 65-18, "Differential Thermal Analysis of Twelve Shale Samples from the Province of Quebec", by K.E. Bell, Mineral Processing Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa.

**Research Report R 187, "The Composition and Properties of Ceramic Clays and Shales of Quebec", by J.G. Brady and R.S. Dean, Mineral Processing Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa

The first, easternmost location was the proposed pit-site, a flat area from which the overburden had been removed to reveal the underlying red and red-and-black shale, which appeared to be uniformly distributed over the entire exposed area, although obviously heavily folded and fractured. Probable locations for drill holes were discussed. A short distance to the west, apparently identical shale was exposed to a depth of about 6 to 8 feet in a pit from which material had been removed primarily for road-fill. However, some of the red shale had been trucked to the plant at Deschaillons where it had been successfully used in a 1:1 mixture with the local clay for the manufacture of flue lining. In addition, a small quantity had been dried and pulverized and was being evaluated for use in an earthenware body at a new pottery at Magog, Quebec. The principal distinguishable material in the pit was plain red shale, although there were substantial quantities of what appeared to be the same material, only blackened on the fracture surfaces. The black or gun-metal discolouration appeared in some instances as a superficial surface stain, in other cases it penetrated to depths of 2 to 3 inches into the blocks of red shale. There were, also, rare 2- to 4-inch seams of very hard black material showing highly polished slickensides. Although the beds were much folded and contorted, and heavily fractured, the general inclination appeared to be about 60° to 70° to the horizontal, dipping towards the north. In a trench at the north end of the pit, apparently over-lying the red shale, was an area of hard grey shale, containing many thin, very hard sandy seams: none of this material was evident at the proposed development site. Samples, weighing between 20 and 40 pounds and representative of the four discernable types of material, were collected and assigned the following identification numbers:

Lab. No. 3000 - Red shale
3001 - Red-and-black shale
3002 - Hard, black shale
3003 - Grey shale

The results of the examination of these samples form Part I of this report.

Subsequently, the companies had two 50-foot holes drilled at the proposed pit-site and the cores were brought to Ottawa for testing for uniformity. Because there were no obvious natural discontinuities in the cores, they were arbitrarily divided at 10-foot incremental depths and assigned the following identification numbers:

Lab. No. 3011 - hole D-1	Lab. No. 3012 - hole D-2
3011A - 0' - 10'	3012A - 0' - 10'
3011B - 10' - 20'	3012B - 10' - 20'
3011C - 20' - 30'	3012C - 20 -- 30'
3011D - 30' - 40'	3012D - 30' - 40'
3011E - 40' - 50'7"	3012E - 40' - 50'5"

The results of the tests performed on these samples form Part II of this report.

PART I

A: Preliminary Examination of Surface Samples

Notes on Hardness

The samples were oven-dried at 105°C (221°F) and ground to pass at least 98 per cent through a 10 mesh screen (Tyler Standard). Stage grinding was employed, with intermittent removal of fines by screening, to simulate commercial grinding results. All of the samples were hard and processing them caused excessive wear on the grinding plates of the small attrition-type grinder employed. Most of the wear was attributed to the grey material, Lab. No. 3003, the sandstone seams probably being the most abrasive. The black shale, sample No. 3002, seemed to be brittle and would probably be more amenable to grinding by impact.

Thermal Analysis

Representative aliquots of the samples were further ground to minus 100 mesh, using an agate mortar, and were examined by differential thermal analysis (DTA), and by thermogravimetric analysis (TGA) where considered useful. The thermograms are shown in Figure 1. The curves for the red and the red-and-black samples are similar and agree closely with those reported previously for St-Apollinaire shale.

In comparison, the curve for the grey sample (No. 3003) shows smaller peaks for adsorbed water at about 200° and 300°C and a smaller main clay dehydroxyllation peak at a slightly lower temperature of about 560°C: this indicates a lesser amount of total clay substance and probably a difference in proportion if not in nature of the clay mineral composition. A small, sharp exothermic-endothermic doublet at just above 400°C results from a minor amount of pyritic material: some of these minerals were visible in finely disseminated form on some of the fracture planes of the sample. The amount is small, and no firing problems would be anticipated, particularly inasmuch as the thermogram does not indicate the presence of carbon compounds. There is, however, some calcite present, indicated by the endothermic peak at about 700°C. All of the samples contained some chalky white material in some of the fracture planes (probably of secondary derivation) that effervesced with dilute HCl. The quantity of this material in the other three samples was so small that it was completely obscured when diluted in the bulk of the ground sample, but ground and extruded samples of the grey shale effervesced rather strongly. The weight loss circa 700°C corresponded to a calcite content of about 3 per cent.

The thermogram of sample No. 3002 (black shale) is complicated by two simultaneous reactions that mutually interfere. A broad exothermic reaction (indicated by thermogravimetric studies to be burn-out of carbon) between about

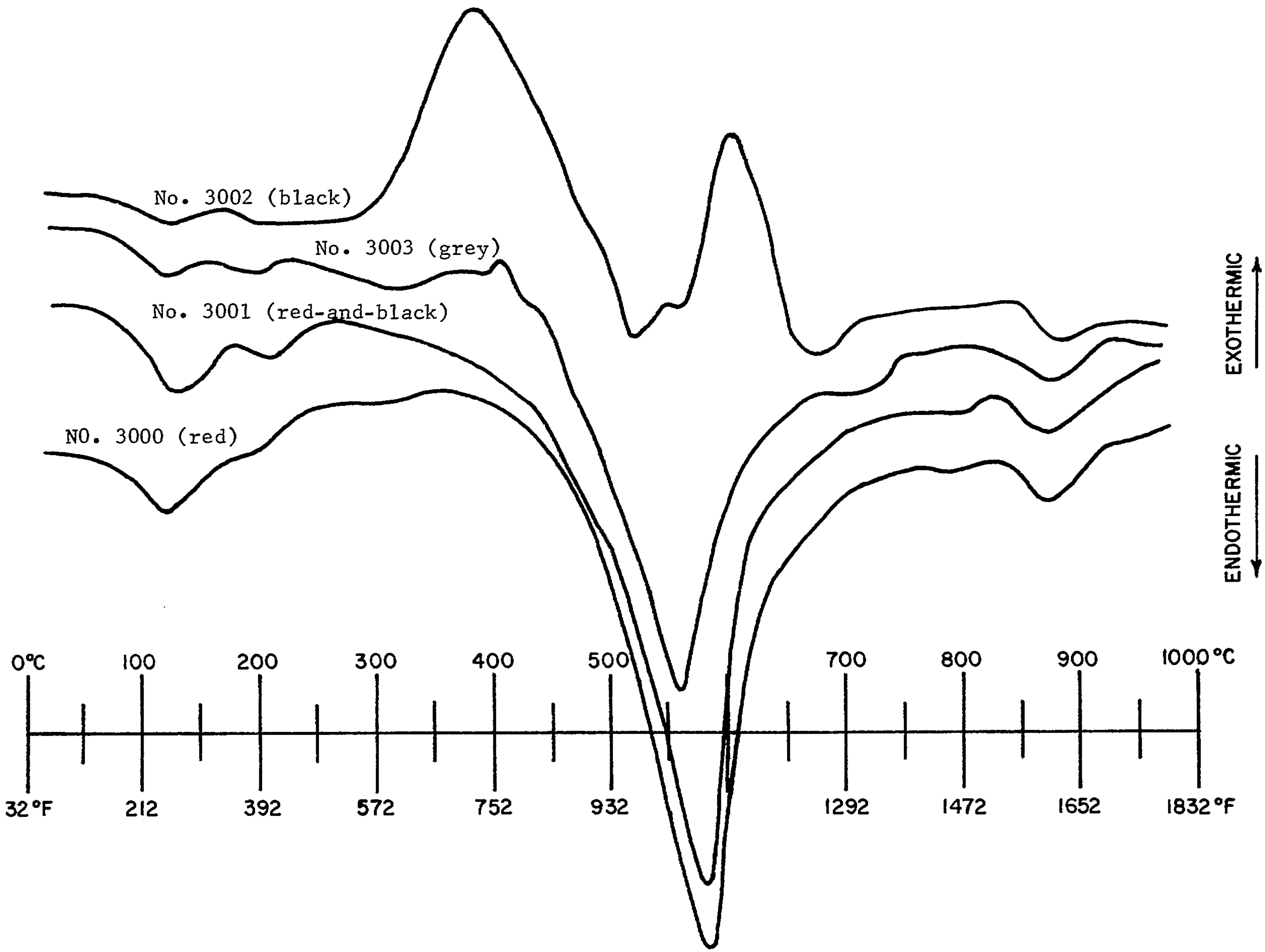

Figure 1. Thermograms of surface samples of shale from St-Apollinaire, P.Q.

300°C and 650°C is superimposed on the endothermic reaction accompanying breakdown of the clay minerals indicated by a double peak above and below 550°C. The double clay peak probably derives from two different clay minerals, likely illite and chlorite. The sample appears to contain a considerable proportion of coal, which could cause oxidation problems: fortunately, the material occurs only in rare, thin seams constituting a nearly negligible proportion of the aggregate deposit.

Plastic Properties

The ground samples were examined, using the Brabender Plastograph. Normal procedures were employed: sample weight, 200 grams; low speed; normal sensitivity; rate of water addition, 1 cm^3/minute. The plastograms are shown in Figure 2. Extrusion tests were made for each sample at water contents just below the indicated peak values: 1 x 1 x 8-inch bars were extruded at full vacuum (28 in. of Hg), using a Loomis hydraulic extrusion press. One freshly formed bar was inserted directly into a hot dry oven maintained at 85°C (185°F), the remainder were air-dried for 24 hours, then oven-dried at 105°C (221°F). Moduli of rupture were determined in 3-point loading, on 3-inch centres, averaging a minimum of 6 specimens.

The plastograms of the red and the red-and-black samples (Nos. 3000 and 3001) are similar: the curves show wide, rounded peaks at about 800 metre-grams of torque, which experience indicates to be minimally adequate for extrusion of simple tile shapes such as pipe or flue-liners. The greater width of the curve for the red-and-black shale, and the higher water requirement for equal consistency, may indicate a greater tendency to slake during pugging. Both samples extruded very well with 12.5 to 13 per cent water; smooth, stiff-plastic columns were obtained with reasonable power consumption. There was no indication

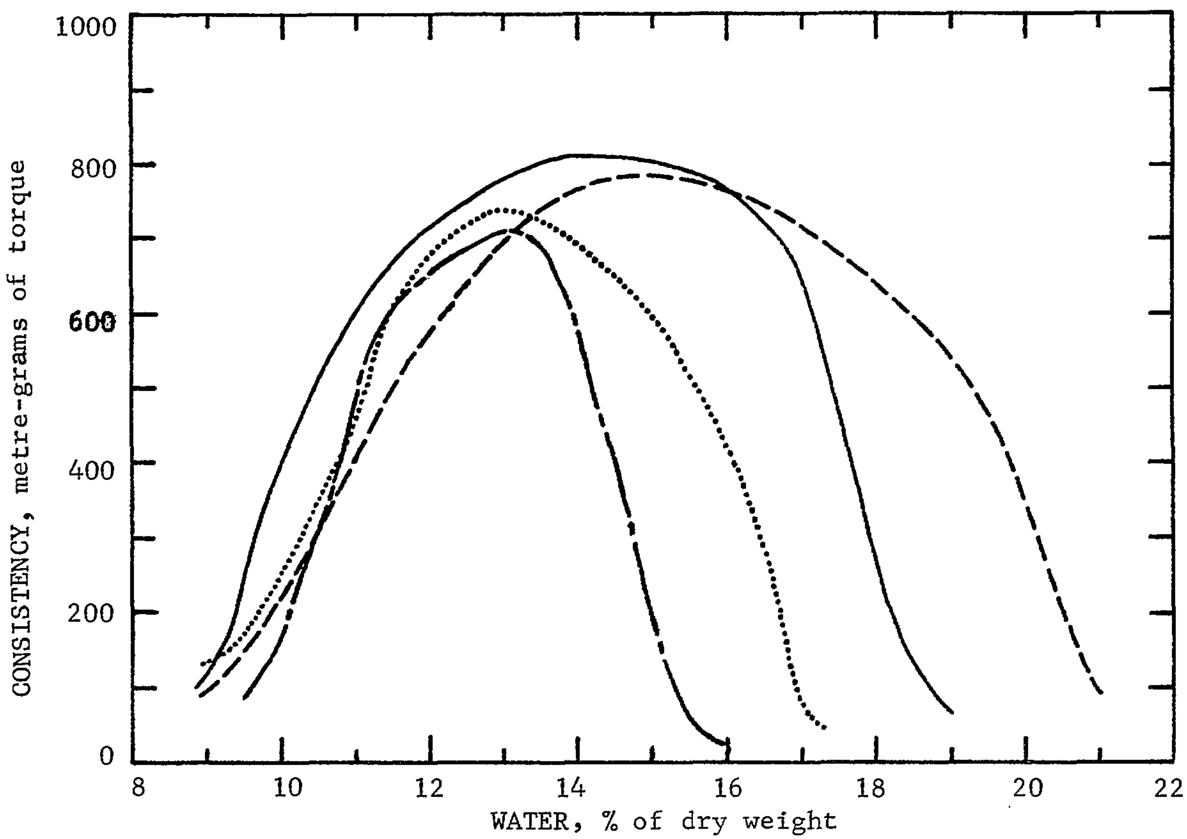

Figure 2. Plastograms of samples of shale from St-Apollinaire, P.Q.

No. 3000 (red) ————

No. 3001 (red-and-black) — — — —

No. 3002 (black) ——— — ———

No. 3003 (grey) ··················

that stiffer columns would be unsatisfactory. The red shale showed a slight sensitivity to severe drying procedures, but neither sample cracked in controlled drying conditions. Drying shrinkages were low: 1.6% for sample No. 3000 (12.5% water) and 1.9% for No. 3001 (13% water). Moduli of rupture were relatively low: 185 psi for sample No. 3000 and 205 psi for No. 3001. Minimum values of twice this magnitude are normal for plastic clays.

Samples No. 3002 (black) and No. 3003 (grey), on the other hand, show rather sharp plastogram peaks at 700 to 750 metre-grams of torque. They appear to contain lesser amounts of clay substance in the first instance, and, because of their hardness, they may have produced fewer plastic fines during grinding. In the black shale particularly, the plasticity of the clay minerals may have been adversely affected by metamorphosis accompanying the folding of the beds. Both samples extruded smoothly at 12% water content, but the power required was about 125 per cent of that for the red shales. They both behaved in a dilatent manner, with bleeding behind the die (most conspicuous with the grey shale). The columns were noticeably less plastic, tending to be "mushy". The specimens dried without cracking, even under severe conditions. Drying shrinkages were very low; 0.9% for the black shale, 0.7% for the grey sample. Dry strengths were considered too low to allow for safe handling of large ware: moduli of rupture values were only 108 psi for the black shale and 94 psi for the grey material.

Fired Properties

Duplicate specimens of each sample were fired in electrically heated furnaces to each of cones 06, 05, 02 and 3. Linear firing shrinkages were calculated in per cent of wet length, so they are directly additive to the previously reported drying shrinkages. Water absorptions were determined on both the 24-hour cold soak and 5-hour boil bases, and saturation coefficients were calculated. Variations of shrinkage and absorption with firing temperature are shown for samples No. 3000 and No. 3001 in Figure 3, and for samples No. 3002 and No. 3003 in Figure 4. All four samples are red-burning, the poorest colour being shown by the grey shale, probably owing to its higher lime content.

The two red materials show wide firing ranges, but begin to overfire before reaching complete densification: minimum absorptions of about 4 to 5 per cent are reached at about cone 01. The rate of overfiring above this temperature is low, which provides a factor of safety, but at cone 3 the surfaces of the specimens were well vitrified and lightly stuck together. More importantly, at cone 3 the specimens sagged slightly under their own weight, which of course would be catastrophic for large shapes such as pipe or flues. At the lower firing temperatures, saturation coefficients are high, ranging from about 0.80 to 0.85, but at cone 02 the values are 0.67 and 0.58 for the red and red-and-black samples, respectively.

The two harder samples, Nos. 3002 and 3003, are more refractory, and just approach the upper limit of desirable absorption when fired at cone 3. Although not apparently highly vitrified, the specimens of the black shale sagged slightly under their own weight at cone 3, and may in fact be at the point of over-firing: the high porosity could be the result of carbon burn-out. At the lower firing temperatures saturation coefficients are high, about 0.9, reaching about 0.8 at cone 02 and 0.7 at cone 3. The grey shale is not sufficiently vitrified in the temperature range studied. Coincident with its high absorption values, saturation coefficients decrease with firing temperature from about 0.9 to only 0.77.

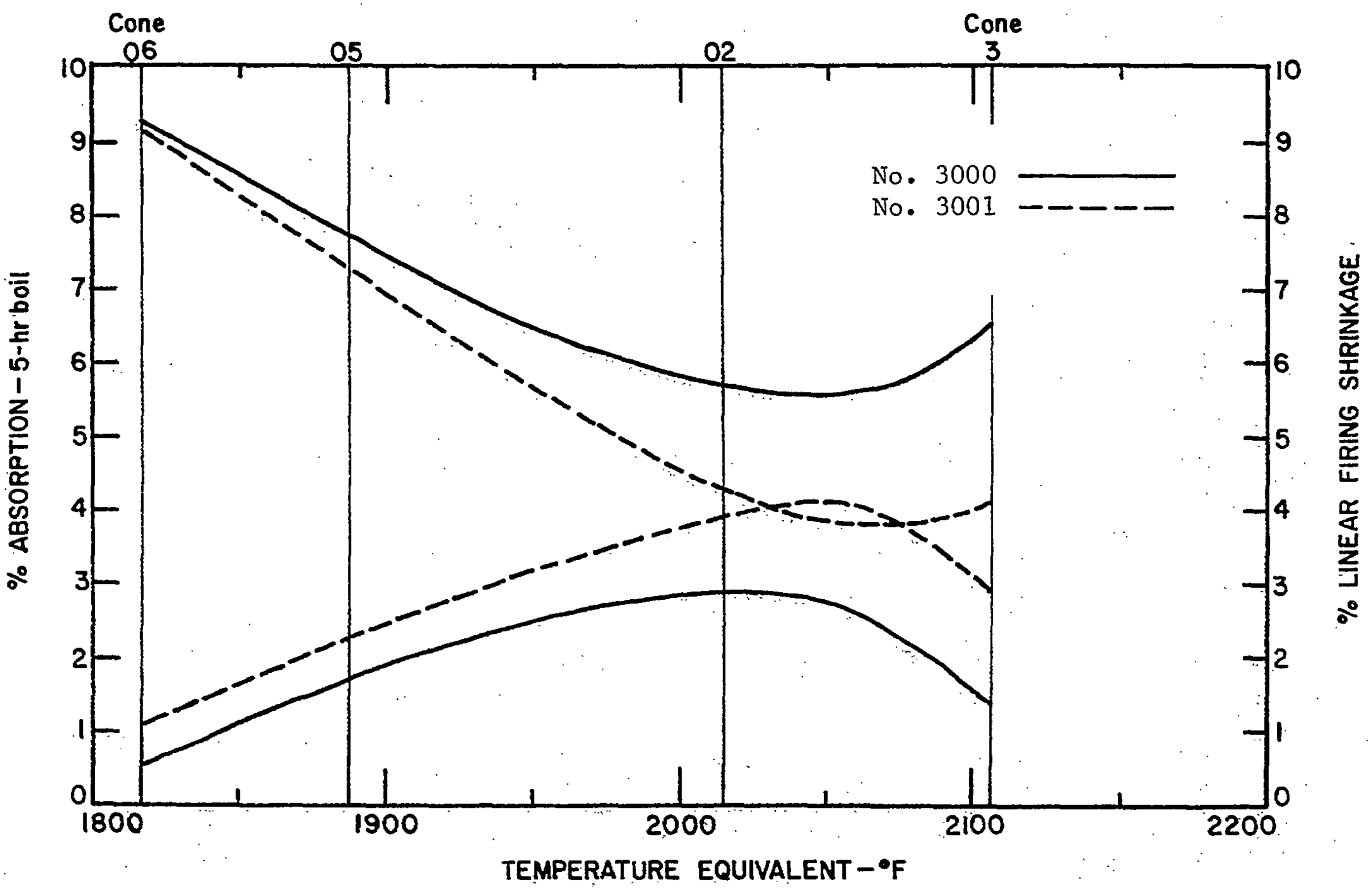

Figure 3. Fired properties of shale samples No. 3000 (red) and No. 3001 (red-and-black) from St-Apollinaire, P.Q.

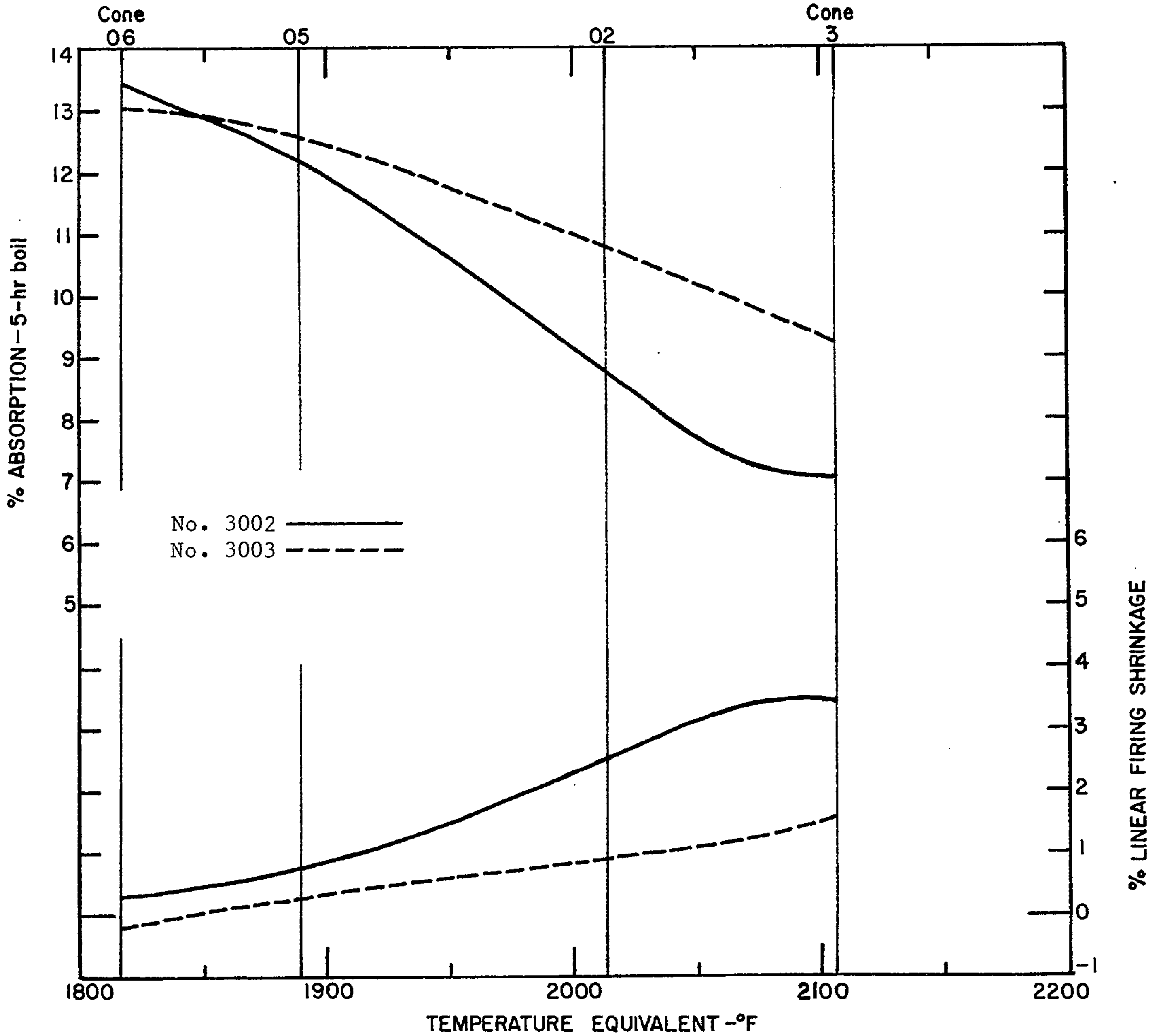

Figure 4. Fired properties of shale samples No. 3002 (black) and No. 3003 (grey).

Summary

Samples No. 3000 and No. 3001, the red and red-and-black shales respectively, show excellent fired properties suitable for sewer pipe and/or flue lining through a wide firing range of temperatures normal to the industry. Their plasticities are probably minimal for extrusion of these types of ware and dry strengths may be lower than desired: they would probably benefit from finer grinding (which may occur in commercial practice) and/or from additives to improve plasticity and dry strength.

From observations at the site, it seems impossible to avoid inclusion of the black shale, sample No. 3002. The indications are, however, that it will make up such a small part of the total that its effect should be negligible.

The grey shale, sample No. 3003, on the other hand, is clearly separable from the main red deposit by selective mining and is in fact not evident at the proposed pit-site. Because of its abrasiveness and its poor plastic and firing properties, it would seem advisable to avoid it completely, although a controlled addition might extend the firing range of the red shales, in the unlikely event such should prove necessary.

B: Shale-Clay Mixtures

Plant tests on the shale from St-Apollinaire, alone and in mixtures with Dundas shale, were conducted at the Ontario plants of National Sewer Pipe Limited and a subsidiary, Canada Vitrified Products Limited. Extrusion difficulties were reported, the nature of which indicated the desirability of increasing the plasticity of the shale. A suitable plastic clay is available at Deschaillons and had already been successfully used in a mixture of 50 per cent shale, 50 per cent clay. Studies were undertaken to determine the effects of lesser clay additions. A sample of clay, No. 2826, representative of the upper 35 feet of the deposit at the Deschaillons plant, was on hand from a previous investigation. This material was used in combination with sample No. 3000, the red shale sample from St-Apollinaire, to form mixtures incorporating 10, 20 and 30 per cent of clay, designated Nos. 3000A, 3000B and 3000C respectively.

Plastic Properties

The mixtures were examined using the Brabender Plastograph. The same procedures previously used were employed. The plastograms are shown in Figure 5, compared with that of the shale alone. For each incremental increase in clay content the maximum consistency is increased by about 100 metre-grams of torque. The water requirement is also increased by about one per cent for each successive increase in clay content.

Each mixture was pugged to a stiff-plastic condition in a Hobart mixer, and a 1-inch square column was extruded under full vacuum, using the hydraulic extrusion press. In each case, the amount of plasticizing water corresponded closely to the point on the plastogram where the rising curve begins to break, considered to be the optimum water content for smooth, stiff extrusion. Some difficulty was experienced in judging this optimum water content for mixture No. 3000C (30% clay). At this level of clay addition the plastic fines become dominant, with "balling" of the material in the mixer, resulting in a deceptively "dry" appearance. An operator used to judging the consistency of predominantly shale mixtures might have some initial control problems, but should quickly become adjusted to the changed appearance.

Each of the mixtures extruded well, producing a sound, smooth column with no evident laminations. None of the mixtures appeared to be particularly plastic: all tended to have a rather brittle fracture, but none showed signs of dilatency.

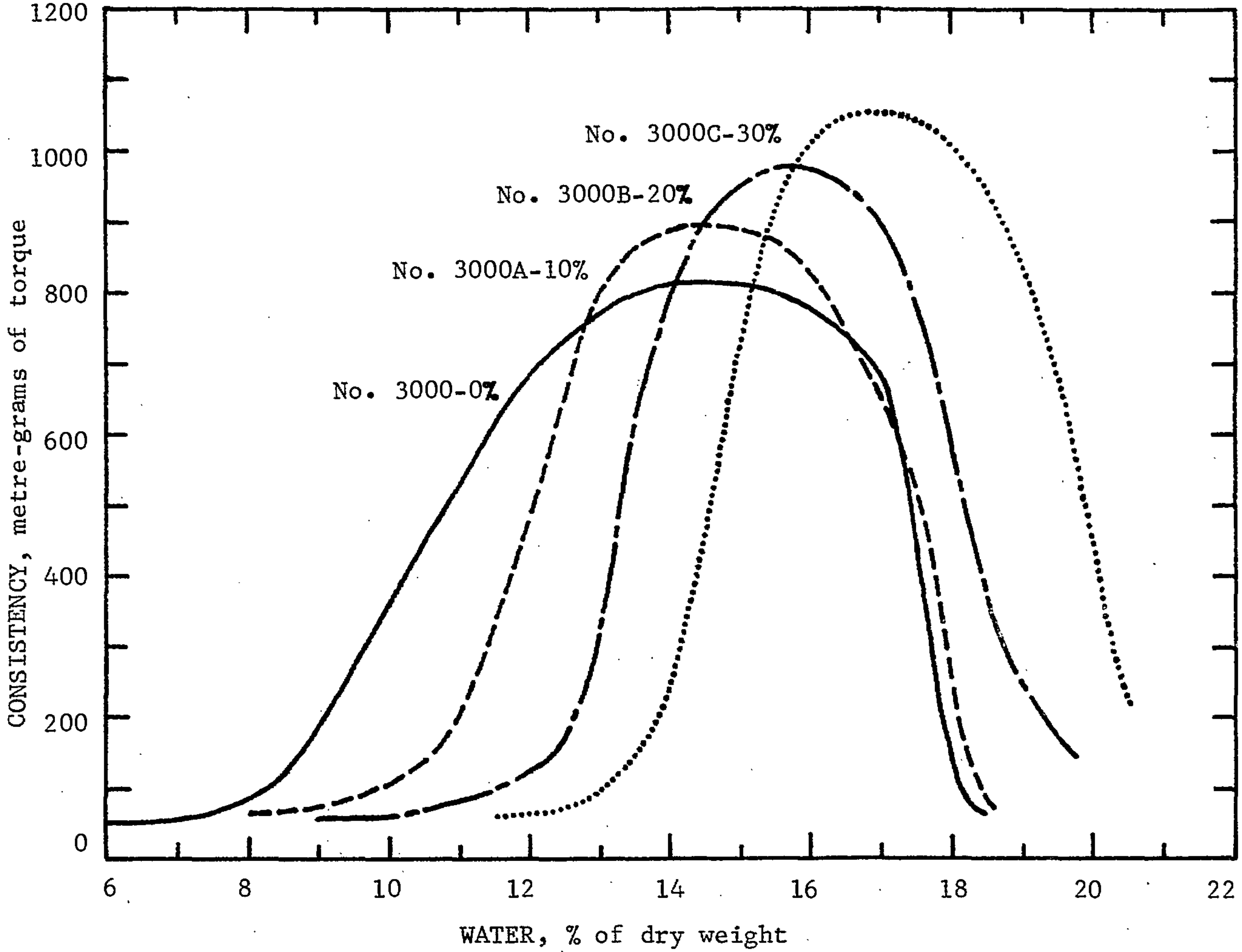

Figure 5. Effect on plasticity of additions of Deschaillons clay No. 2826 to St-Apollinaire shale No. 3000.

All of the mixtures dried without cracking under both severe and slow-drying conditions. Drying shrinkages were low, around 2 per cent. Dry strengths were significantly improved, dry moduli of rupture (3-inch span in 3-point loading) values were 260,354 and 365 psi for the 10,20 and 30 per cent clay mixtures, respectively, compared with the value of 185 psi previously reported for shale No. 3000 alone.

The plastic and dry properties of the mixtures are summarized in the following Table.

TABLE 1

Plastic and Dry Properties of Mixtures of St-Apollinaire Shale and Deschaillons Clay

Sample	Plasticizing Water (% of dry wt)	Pressure for Extrusion (tons)	Drying Shrinkage (% of wet length)	Modulus of Rupture (psi)
No. 3000 (100% shale)	12.5	1.25 - 1.75	1.6	185
No. 3000A (10% clay)	12.5	1.75 - 2.5	1.9	260
No. 3000B (20% clay)	14.25	1.5 - 2.25	2.0	354
No. 3000C (30% clay)	15.4	2.0 - 2.75	2.2	365

Fired Properties

Duplicate specimens (1 x 1 x 4 inches) of each mixture were fired in electrically heated kilns to each of cones 06, 04, 02 and 1. Figure 6 shows the variation in fired properties (shrinkage and absorption) with firing temperature for the mixtures. Comparative data for the shale alone is shown in Figure 3.

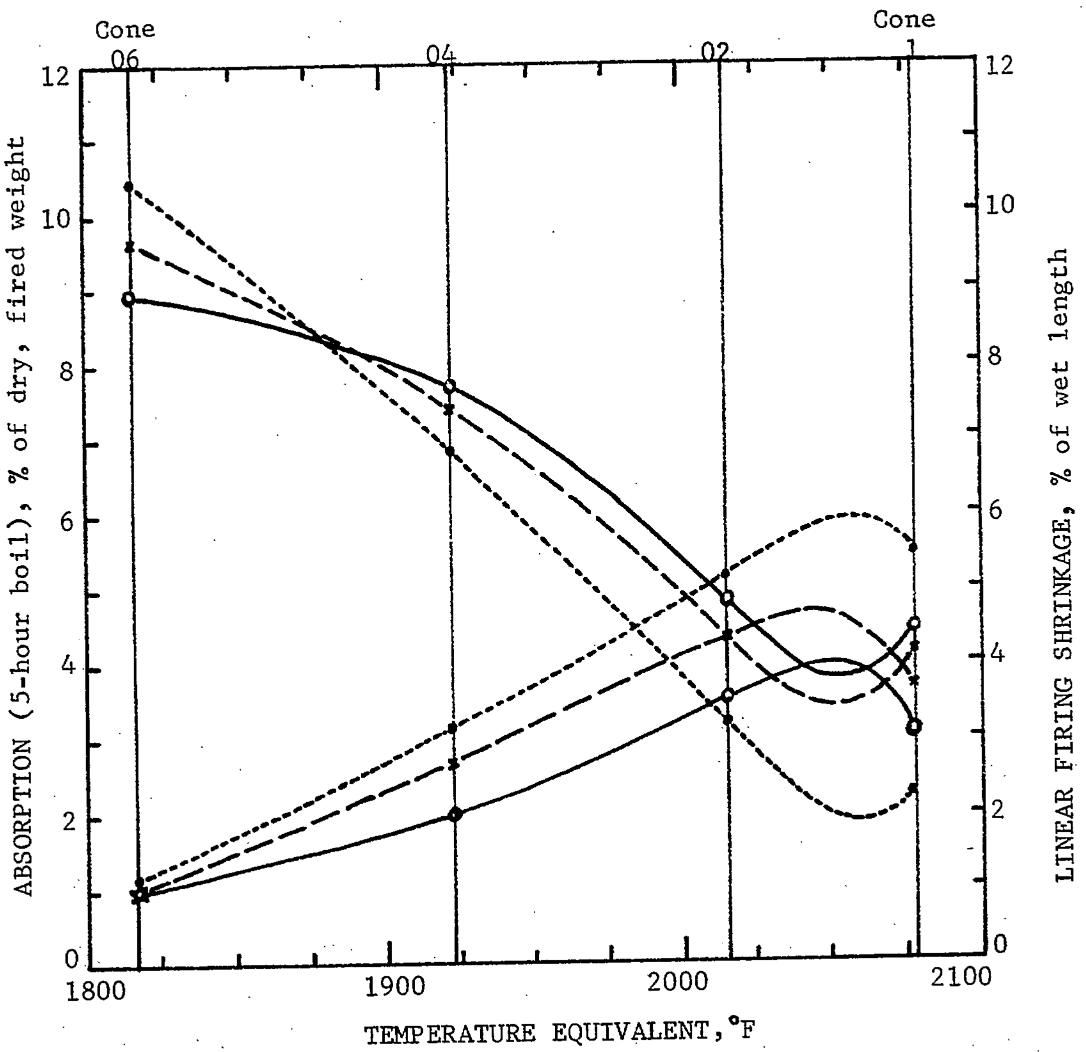

Figure 6. Effect of additions of Deschaillons clay No. 2826 on fired properties of St-Apollinaire shale No. 3000.

90% shale, 10% clay — 80% shale, 20% clay — 70% shale, 30% clay

With increasing clay content, these changes in properties become progressively greater, but remain within acceptable limits. Suitably low absorptions of 8 per cent are reached at about cone 05. At cone 02, the specimens were still of excellent mechanical appearance, indicating safe firing ranges before overfiring in excess of 100 Fahrenheit degrees. (At cone 1 the surfaces of the specimens were vitreous and the bars had begun to deform under their own weight). Within these limits, variations in firing shrinkage did not exceed about 2 per cent, promising good product size control. In addition, the mixtures come closer to complete vitrification before overfiring, promising increased impermeability of products. Saturation coefficients of all three mixtures are marginal - 0.87 to 0.89 - at cone 06, but at cone 04 and higher temperatures they are 0.78 and lower.

Summary

Addition of plastic clay from Deschaillons to the shale from St-Apollinaire improves the plasticity and extrusion behaviour and significantly increases dry strength. Although the firing range is somewhat narrowed, it remains of ample width for good kiln and product quality control. It should be possible to produce pipe and tile of excellent quality from a mixture of, e.g., 25 per cent clay and 75 per cent shale.

PART II

Description of Cores

The cores from the two drill-holes were received, properly boxed, with the depths of each successive section marked on spacer blocks. Because much of the core was badly broken, and in several cases worn from rotating in the core barrel, it was impossible to judge from linear measurements whether or not each section represented the full depth as marked, or the extent of any lost core. Therefore, the cores were thoroughly oven-dried at 110°C (230°F) and representative samples were weighed and measured, to determine a unit weight. Based on the average values from a number of such determinations, the nominal weight of a 10-foot section was calculated for both sizes of cores supplied: as the cores were subsequently examined and divided, each section was weighed and its nominal length was calculated in proportion to its weight. (In the case of the one section consisting of cores of two diameters, the weight of the larger core was reduced in proportion to the diameters of the cores and the excess discarded, to ensure that the sample would be representative of the depth of the section.) The salient visual characteristics of the core sections are given in Tables 2 and 3.

The cores consisted of red shale with no perceptible stratification according to colour or texture. There was, however, a dominant set of fracture planes inclined at about 50° to 60° with the horizontal (assuming vertical drilling) which also correlated with the angle of most of the thin green seams cut by the core. As this angle corresponded to the apparent pitch of the beds as observed at the surface, it was assumed to represent the bedding plane existing prior to folding. This angle was observed through the length of the cores, as any sudden change would probably be indicative of subterranean discontinuities and would signal a possible change in character of the material. No such marked changes were observed: although the dominant angle of fracture did vary between about 45° and 75° with the horizontal, it did so in a regular and progressive fashion, consistent with regular folding throughout the depths of the holes. A secondary system of fractures was clearly discernible, consistently inclined at 85° - 90° and 10° - 15° with the horizontal: these fractures are assumed

TABLE 2

Core Log: Hole Dl, St-Apollinaire (Sample No. 3011)

Depth Drilled	Sample	Description
0-2'3" -3'4" -5'9" -7'1" -9'6"	No. 3011 A Wt= 8,110 g (Estimated lost core = 1'5")	Upper 7 ft of very badly broken and worn core, remainder broken. Red shale, with most fracture planes through the upper 8 ft stained black and rusty; traces of rust stains on remainder. Rare green lenses ranging from 5 mm to 4 cm diam. and 0.5 to 3 mm thick. No apparent white material in upper 8 ft (washed out?), traces on several fractures at bottom of section. Bedding plane fractures varying progressively from 60° - 65° at top to 75° at 5-ft depth, reverting to 50° at bottom of section: secondary fracturing rarely observable at 15° and 80° - 85°.
	10'0"	
-11'1" -13'8" -15'1" -17'5" -19"2'	No. 3011 B Wt=9,300 g (Estimated lost core = 1.5")	Broken to moderately broken core, badly broken and worn at 14'-15' depth. Red shale, with occasional black stained fracture surfaces. Rare green lenses, from 1 to 5 cm diam., from very thin to 2 mm thick; one green seam about 3 mm thick at 15-ft depth; multiple, very thin green seams at 17 to 20 ft. Traces of white deposits on many fractures, heaviest at 11- to 12-ft depth. Bedding-plane fractures at 45° to 50° throughout; much secondary fracturing at 80° - 85° and 10° - 15°, particularly in upper half of section. Aggregates of pyritic minerals observed on one fracture at about 15-ft depth.
	20'0"	
-22'8" -27'2"	No. 3011 C Wt=9,600 g (Estimated 2" extra core)	Mostly good core, broken for about 6" at each recommencement of drilling. Red shale, some black stains on fracture faces. Many small green lenses and spots in upper part of section; green seams common (very thin to 3 mm thick); little green material in bottom 3 ft of section. Traces of white deposits on many of the major fractures. Bedding fractures at 50° to 55°; many fractures

(Continued)

TABLE 2 (continued)

Depth Drilled	Sample	Description
		at 85°, often black-stained. Pyrites common through 28' to 29' depth, not associated with green material.
	30'0"	
-30'10" -35'8" -38'6"	No. 3011 D Wt=9,260 g (Estimated lost core = 2")	Good core. Red shale with minor black staining; 6-in. seams of hard black shale at 37½' and 39'. Rare green lenses and thin seams, one major seam (4 to 5 mm thick) in each half of section. Minor white deposits on fracture planes of upper 4 ft of section, becoming thicker (0.5 mm) and flaky through bottom of section: heavy deposit (5 mm thick) in major fissure at 34½' depth. Bedding fractures rarely apparent, but vary from 50° at top to 60° at bottom of section: secondary fractures at 10° to 15° and 85° to 90°, one markedly slickensided. Pyritic minerals visible in black shale seams.
	40'0"	
-42'11" -47'1" -50'7" End	No. 3011 E Wt=9,970 g (Estimated ½" extra core)	Good core. Red shale, with thin (2 to 3 cm) seams of hard black shale at 45' and 47½'. Upper 3 ft of section contains multiple green seams 2 to 4 mm thick, becoming thin in centre of section and very thin and rare in bottom 3 ft. Bedding fractures not apparent, but inclination of green seams varies progressively from 60° at top to 40° at bottom of section; core tends to split lengthwise (at 90°) with traces of white deposit on interior planes, some of which are slickensided. Pyritic aggregates evident in red shale at about 49½' depth.

TABLE 3

Core Log: Hole D-2, St-Apollinaire (Sample No. 3012)

Depth Drilled	Sample	Description
0-1'10" -4'0" -6'2" -8'7"	No. 3012 A Wt=6,900 g (Estimated lost core=32")	Broken core: top 6 ft particularly badly broken and worn. Red shale, rust-stained, black staining on most open fractures, decreasing towards bottom of section. Rare green lenses ranging from 5 to 25 mm diameter and from very thin to 3 mm thick; very rare, thin seams; green material noted along several vertical fractures. Traces of white deposits on fracture planes throughout, becoming heavier but spotty at bottom of section. Inclination of bedding planes about 40° at top, 50° at bottom of section; bottom half of section shows frequent fractures at 85°.
	10'0"	
-11'2" -15'4" -19'2"	No. 3012 B Wt=10,170 g (Estimated 9½" extra core)	Mostly good core: top 1 ft badly broken. Red shale, rarely rust-stained, little black staining. Rare green material in upper and lower parts of the section, heavy concentration of thin green lenses and seams at 12- to 14-ft depth. White deposits rare in upper half of section, heavy on many fractures in lower half. Bedding inclination constant at about 45° throughout section, although not well-defined in solid core sections. Pyritic aggregates smeared on 2 slickensided near-vertical fractures.
	20'0"	
-22'0"	No. 3012 C Wt=10,030 g (Estimated 7½" extra core)	Mostly good core: badly broken and crushed between 24' and 25'. Red shale, minor black staining at middle of section only. Minor thin green seams, rare small lenses in upper portion; frequent, very thin green seams at 80° in bottom 4 ft. Heavy deposits of white material on open fractures, 0.5 to 1 mm thick, blotchy and flaky; thin, spotty white deposits on many fresh fractures, usually accompanied by much visible pyrites on near-vertical (slickensided) fractures. Bedding

(Continued)

TABLE 3 (continued)

Depth Drilled	Sample	Description
		fractures rare, varying from 40° to 45° at top to about 55° at bottom of section. Much splitting at 80° to 85° and at 10°, vertical planes usually slickensided.
	30'0"	
-31'2" -32'5" End large core	No. 3012 D Wt large core= 2,420 g (Estimated $1\frac{1}{2}$" extra core) Wt small core = 2,660 g (Estimated lost core=$44\frac{1}{2}$")	Large core good to broken: only about 2 ft of good small core, remainder badly broken. Red shale with black staining and traces of black on fractures throughout the section. Bedding fractures rarely discernible; indicated at 45° at top, increasing to 55° to 65° at bottom of section. Multiple green seams at top and bottom 2 ft of section, thick (3 to 4 cm) at top, very thin at bottom; rare, very thin seams in central part of section. Heavy white deposits (1 to 2 cm) on near-vertical planes in upper 2 ft of section, becoming thinner with increasing depth. Fractures at 80° to 90° occasionally slickensided, rarely show traces of pyrites.
	40'0"	
-41'0" -42'8" -48'8" -50'5" End	No. 3012 E Wt=4,110 g (Estimated lost core=53")	Good core in central 3 ft of section only, remainder badly broken, making fracture angles difficult to determine. Bedding fractures appear to be 60° to 65° throughout section; much fracturing at 15°. Many small green lenses and thin seams; two thick seams (4 to 5 cm) at centre of section. White deposits heavy and common at top of section, rare and only on internal faces in bottom 7 ft (washed out by drilling?). Pyrites visible on most red and green fracture planes in bottom half of section.

to represent two vectors of shear accompanying folding. This assumption is reinforced by the fact that a number of the near-vertical fractures were slickensided and that instances were observed of displacement of the green seams along these planes.

The cores frequently cut these thin seams of greyish green material, which ranged in thickness from about 1 to 6 mm. Except for a few of the very thinest seams, these represented planes of weakness along which the core could be readily split. In addition to the "seams" of green material (which extend over the full cross-section of the core but which might, in reality, be large lenses) there were a multiplicity of small lenses of greenish material ranging in size from a few mm to 5 or 6 cm in diameter and in thickness from about 0.5 mm to 3 to 4 mm. In many instances, these green lenses were seen to be associated with slightly open cracks, usually aligned with the dominant (basal) fracture planes: only in 3 or 4 cases throughout the 100 feet of core was greenish material noted on the near-vertical (secondary) set of fracture planes. A thin-section through the red and green materials was prepared and examined microscopically in the Ore Mineralogy section: no differences other than colour were observed. X-ray diffraction analysis of the greenish material revealed only quartz and clay minerals.

The small green lenses and the thinner seams, at least, invariably occluded aggregates of pyritic minerals. Pyrites were also frequently observed on red fracture planes, primarily on those parallel to the bedding fracture. On two occasions where pyritic aggregations were visible on near-vertical fractures, they appeared to be smeared, indicative of vertical movement having taken place after deposition.

Many fissures and cracks were filled or coated with a whitish material, apparently of more recent, secondary, deposition. In thicknesses ranging up to about 5 mm it had a chalky appearance, but in thin coatings it had a vitreous (quartz-like) lustre. When treated with dilute HCl, it effervesced only moderately. X-ray diffraction analysis revealed that it consisted principally of silica, with small amounts of calcite and dolomite. The facts that it is only a minor constituent, and that the carbonate content is low, explain why no calcite or dolomite peaks were observed on the DTA curves for the samples of red shale reported in Part I.

The ten samples derived from the cores were crushed separately in a laboratory jaw-crusher to about 4 mesh maximum size. Representative aliquots were obtained, by riffling, for a number of tests designed primarily to establish the degree of uniformity between holes and with depth of the deposit.

Differential Thermal Analysis (DTA)

Representative aliquots of each core sample were ground to minus 100 mesh in an agate mortar and examined by DTA. The thermograms are shown in Figures 7 and 8.

The curves for the 10 samples are characterized to a much greater extent by their strong similarities than by their minor differences. The main difference between the two sets of curves is that the main dehydroxyllation peak at about 600°C is longer and slimmer for the No. 3012 series samples (Hole D-2). This probably reflects some difference in proportions of the clay mineral complexes making up the samples, but little difference in total clay content, which is considered to be proportional to the area included by the curve. There are indications, too, in the sizes of the twin peaks at about 100° to 200°C, of slight differences in clay mineral composition with depth in the deposit, probably owing to more-intensive weathering nearer the surface. The broad exothermic peaks occurring in some curves between about 250° and 350°C are probably due to small amounts of carbon-bearing black shale. Oxidation is

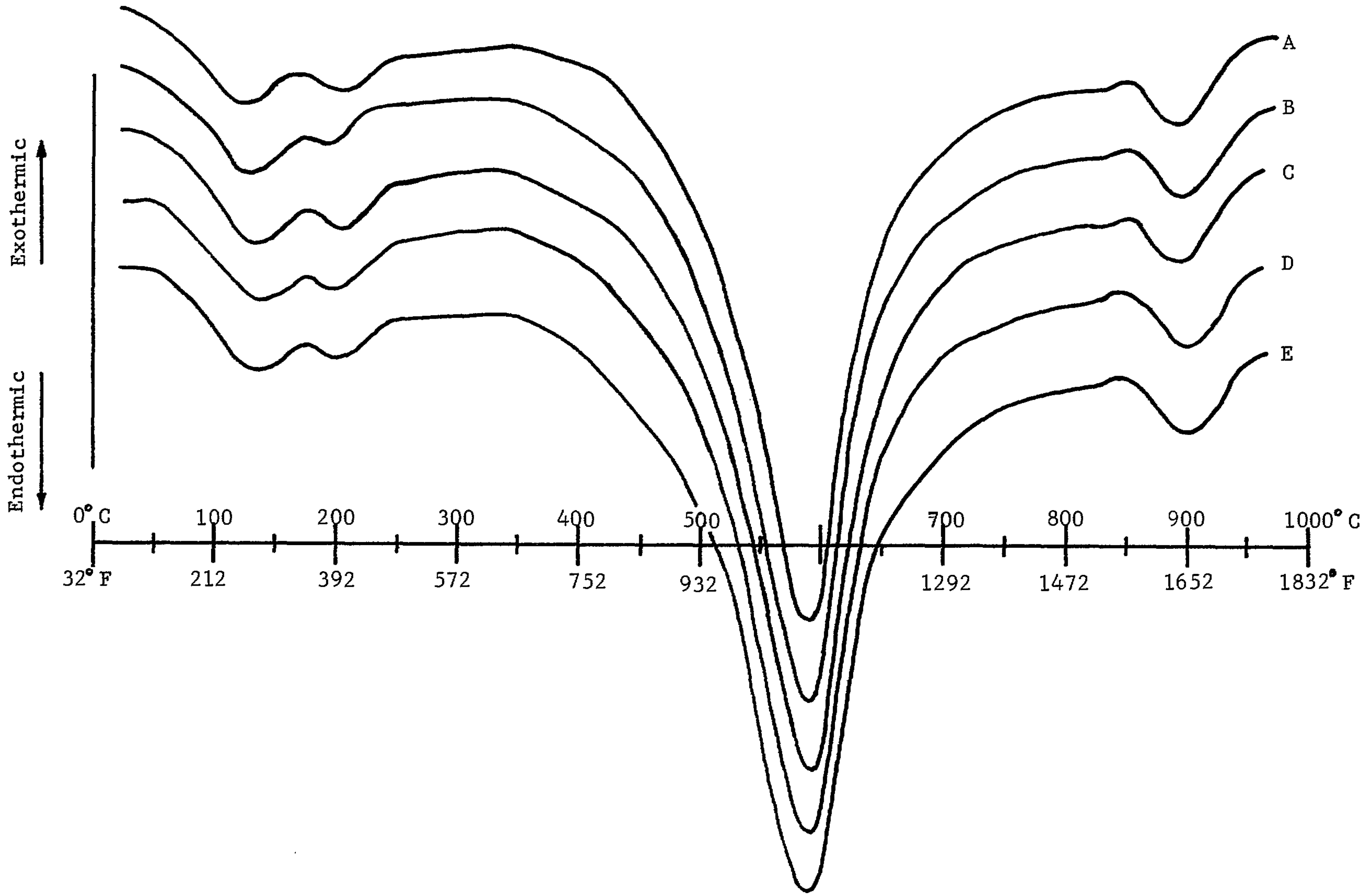

Figure 7. Thermograms of shale samples from Hole D-1, St-Apollinaire, P.Q. (No. 3011 series): A = 0'-10' core section; B = 10'-20'; C = 20'-30'; D = 30'-40'; E = 40'-50'7".

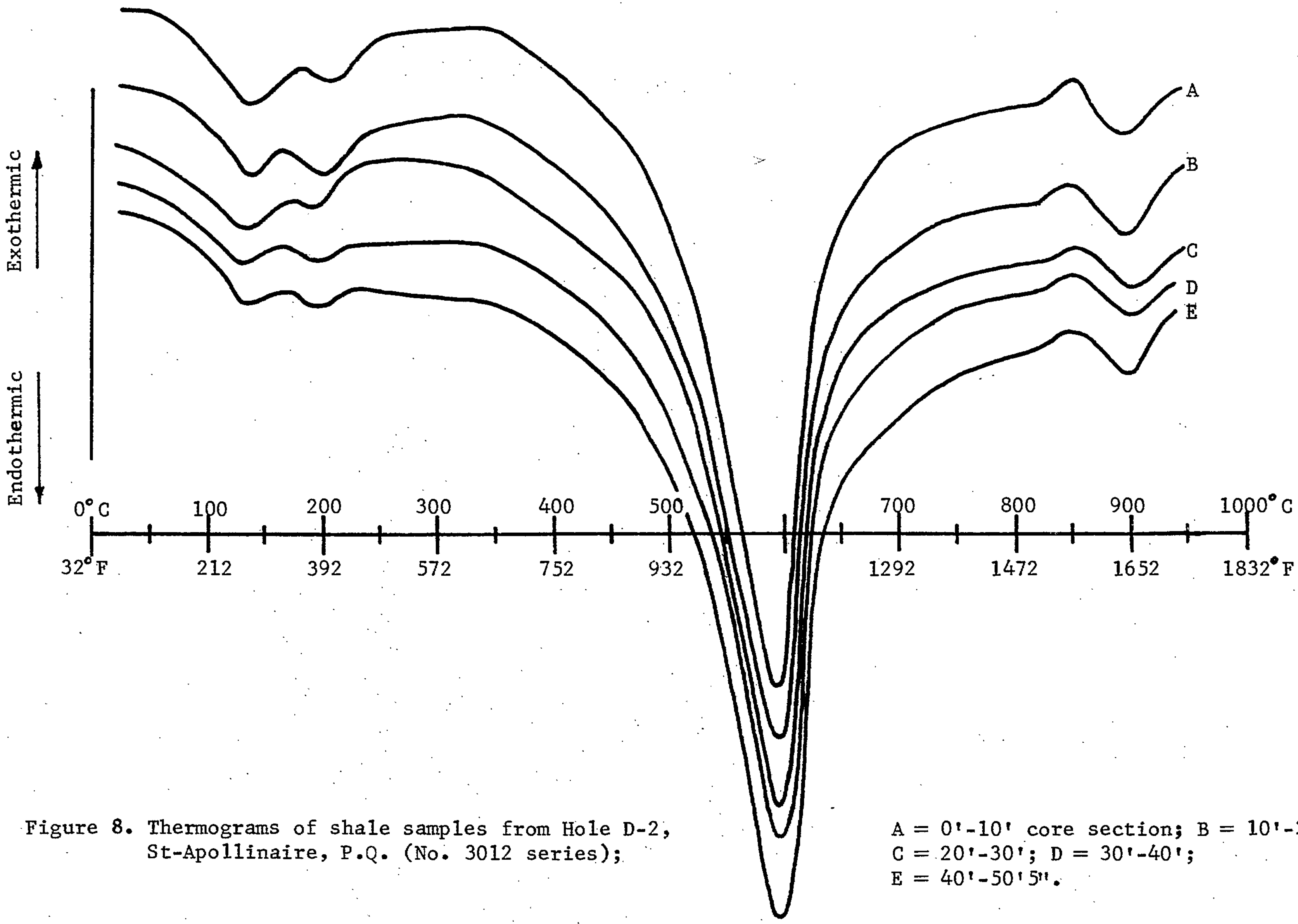

Figure 8. Thermograms of shale samples from Hole D-2, St-Apollinaire, P.Q. (No. 3012 series); A = 0'-10' core section; B = 10'-20'; C = 20'-30'; D = 30'-40'; E = 40'-50'5".

unlikely to be a problem, particularly as all indications are that the hard black shale occurs only in rare, thin seams. The curves show no peaks indicative of pyritic minerals, normally characterized by peaks at about 425°C. The total content of these minerals must be too small to register a detectable thermal effect: probably less than about 0.1 weight per cent of FeS_2.

Plasticity

Representative aliquots of the samples were ground to pass a 10-mesh Tyler Standard screen, employing stage-grinding procedures. The samples were fairly hard and appeared to increase in hardness with depth.

Two-hundred-gram portions of the samples were extracted by riffling for examination using the Brabender Plastograph: test parameters were the same as previously detailed (Part I). The plastograms are shown in Figures 9 and 10. At the conclusion of the tests, a small, but perceptible amount of wear was observed on the blades of the mixing chamber of the instrument, indicative of the hardness and abrasive nature of the material.

The curves for the samples representing the upper sections of both holes (3011A, 3012A) are closely similar to that for the red-and-black surface sample reported previously (No. 3001, Figure 2). With increasing depth of both holes, the curves become narrower, although the peak heights remain about the same. This behaviour is consistent with increasing hardness with depth, resulting in differences in grain size distribution from grinding and in the production of fewer plastic fines during pugging of the harder samples.

Extrusion Behaviour

The ground samples were pugged to a stiff-plastic condition in a Hobart mixer. The water content generally accorded with the break in the rising plastograph curve for each sample but, for the harder samples from the

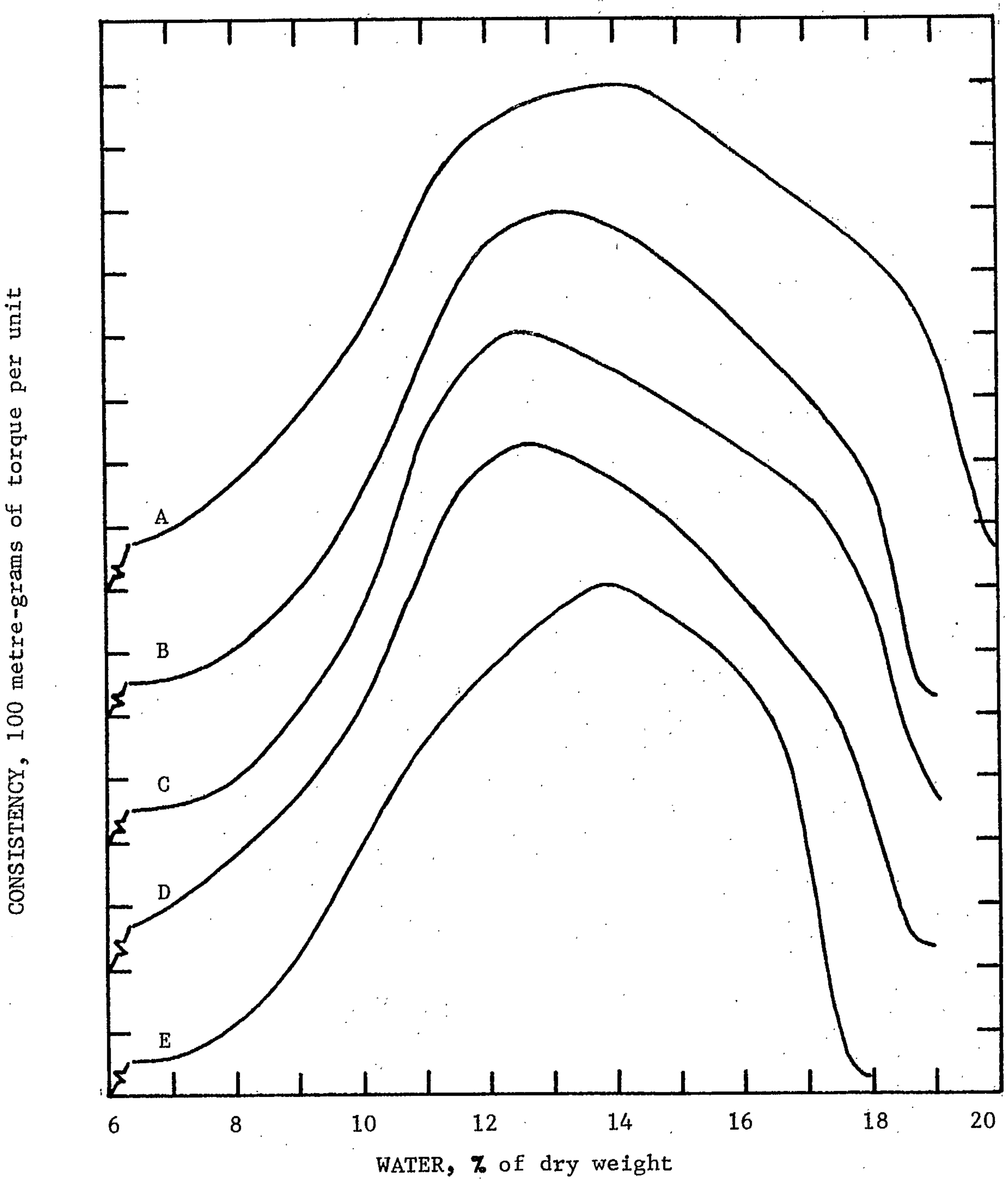

Figure 9. Plastograms of shale samples from Hole D-1, St-Apollinaire, P.Q. (No. 3011 series): A = 0'-10'; B = 10'-20'; C = 20'-30'; D = 30'-40'; E = 40'-50'7".

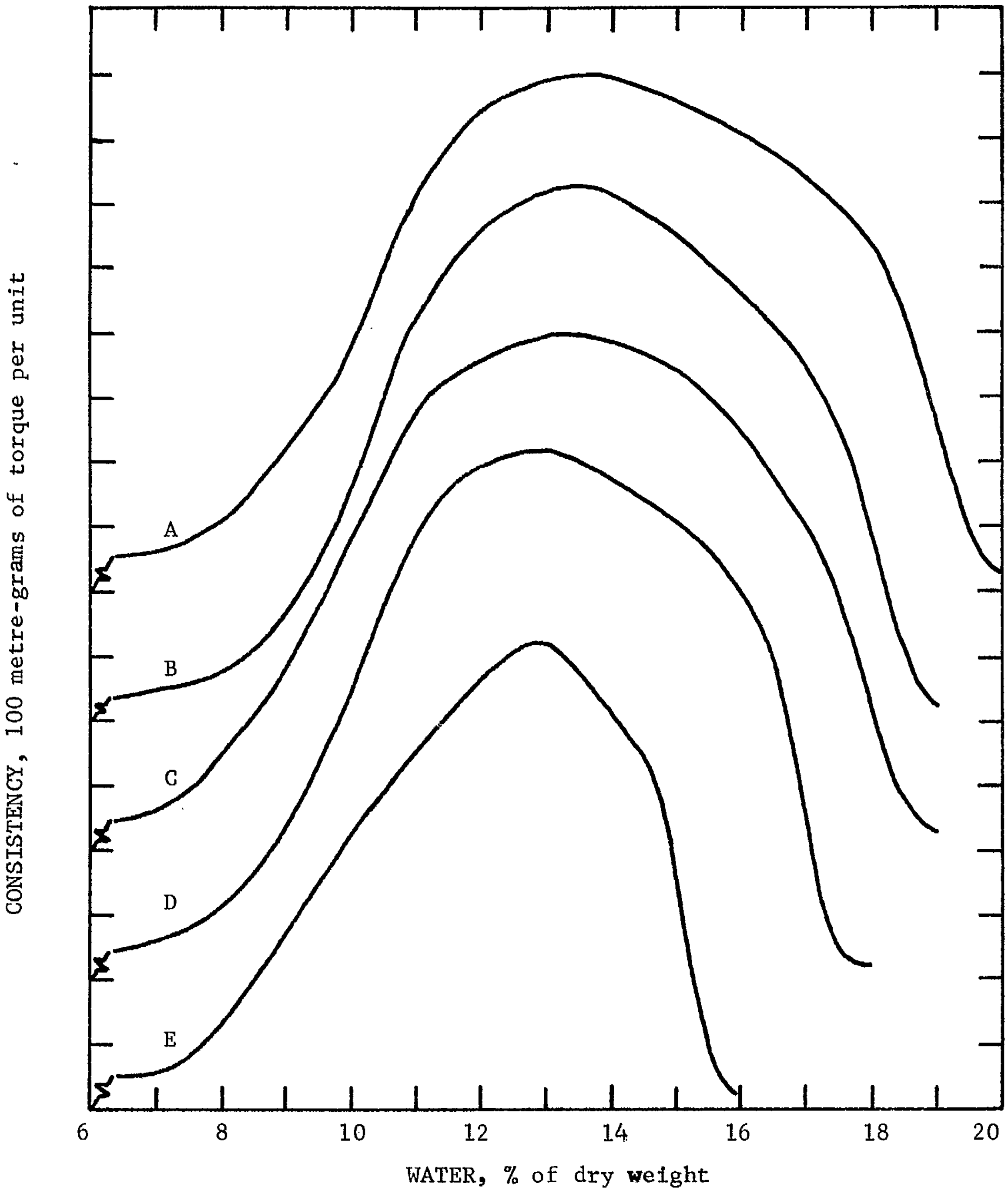

Figure 10. Plastograms of shale samples from Hole D-2, St-Apollinaire, P.Q. (No. 3012 series): A = 0'-10'; B = 10'-20'; C = 20'-30'; D = 30'-40'; E = 40'-50'5".

bottoms of the holes in particular, less water was added because the pugged materials appeared wetter.

The samples were extruded under full vacuum, using the Loomis hydraulic extrusion press to form a 1 x 1-inch column. None of the samples from the cores extruded as well as the surface samples of red shale: the latter have probably been subject to more weathering. Although all of the samples produced mechanically sound columns (square and smooth, no tearing at corners), none was particularly plastic; all tended to be weak and a bit brittle. The rate of extrusion was very responsive to the applied pressure, and all of the samples showed traces of "bleeding" behind the die, which became more pronounced with depth of sample. This behaviour is typical of dilatent materials and is no doubt due to a high content of non-plastic grains and of pseudo-plastic fines. The effect would doubtless be accented under the extra shearing of the augers in a commercial extruder: minor variations in moisture content would greatly influence the small content of truly plastic fines, causing large variations in flow rates (shear rates) and in the extent of dilatency, making the column very difficult to control.

All of the samples dried safely, even under severe conditions, and drying shrinkages were uniformly low. Dry moduli of rupture ranged from 106 to 167 psi.

The plastic and dry properties of the samples are summarized in Table 4.

Refractoriness

Standard-size cones were formed in steel moulds from the plastic, de-aired materials as extruded. Pyrometric cone equivalents (PCE's) of the samples, relative to Orton Standard cones, were determined in a suitable furnace

TABLE 4

Plastic and Dry Properties of St-Apollinaire Shale from Drill-Core Sections

Sample	Water for Extrusion	Remarks	Dry Shrinkage*	Modulus of Rupture
No. 3011 (Hole D1)				
A, 0' - 10'	12.5%	Fairly stiff, not too strong or plastic. Trace of bleeding. Pressure on extrusion ram 1.75 to 2.5 tons.	1.7%	106 psi
B, 10' - 20'	12.0%	Stiff, but brittle. Considerable bleeding. Pressure 2.25 to 3 tons.	1.3%	115 psi
C, 20' - 30'	11.5%	As B, column speeds very responsive to pressure, 2 to 3 tons.	1.4%	129 psi
D, 30' - 40'	11.0%	Very stiff. Some bleeding. Pressure 3 to 4 tons.	1.3%	111 psi
E, 40' - 50'7"	11.5%	Stiff. Some bleeding. Pressure 2.25 to 3 tons,	1.5%	143 psi
No. 3012 (Hole D2)				
A, 0' - 10'	11.0%	Stiff column, not too plastic. Trace of bleeding. Pressure 2.5 to 3.25 tons.	1.5%	148 psi
B, 10' - 20'	11.5%	Fairly stiff, only moderately plastic. Speed sensitive to pressure, 1.75 to 2.5 tons.	1.7%	167 psi
C, 20' - 30'	11.0%	Stiff. Trace of bleeding. Pressure 2.5 to 3.25 tons.	1.3%	144 psi
D, 30' - 40'	11.0%	Very stiff. Some bleeding. Pressure 3.25 to 4 tons.	1.0%	122 psi
E, 40' - 50'5"	10.5%	Very stiff. Considerable bleeding. Pressure 3.5 to 4.5 tons.	1.0%	167 psi

*Linear shrinkage, % of wet length, directly additive to firing shrinkage.

fired by natural gas, at a heating rate of 60 Celsius degrees (108 Fahrenheit degrees) per hour. The PCE's of all the samples fall between Cone 11 (1294°C, 2361°F) and Cone 12 (1306°C, 2383°F).

Firing Behaviour

Duplicate 1 x 1 x 4-inch extruded bars of each sample were fired in electrically heated kilns to each of cones 06, 04, 02 and 2. Shrinkage measurements were made to the nearest 0.01 inch (0.3% equiv.) and linear firing shrinkages were calculated on the basis of wet (extruded) length. Fired absorptions were determined on both the 24-hour cold soak and 5-hour boil bases and saturation coefficients were calculated. The results, given in Table 5, indicate good uniformity of firing behaviour, both with depth and between the two holes. They are much like those obtained for Sample No. 3001, the red-and-black surface sample, and indicate a good wide firing range below 8 per cent absorption. Overfiring begins about cone 1, well before complete vitrification is obtained. Shrinkages are low, and saturation coefficients are acceptable, particularly in the upper half of the firing range.

Summary

There is a marked degree of uniformity between the materials from the two holes and over their depths. This is evidenced by:

1. Similar appearance: neither the extent of the black staining nor the amount of green material appears to significantly affect the ceramic properties. The white deposits are primarily silica and contain too little lime to affect the behaviour of the major shale component.

2. The DTA curves indicate none but minor differences. The pyrites content is too low to indicate oxidation problems.

TABLE 5

Fired Properties of St-Apollinaire Shale
From Drill-Core Sections

Sample	Cone 06			Cone 04			Cone 02			Cone 2		
	Shr	Abs	C/B	Shr	Abs	C/B	Shr	Abs	C/B	Shr	Abs	C/B
No. 3011 (Hole D1)												
A, 0' - 10'	0.5	9.9	0.82	2.0	7.3	0.77	2.2	6.1	0.73	0.3	6.1	0.63
B, 10' - 20'	0.7	9.2	0.82	1.5	7.9	0.77	2.0	5.9	0.72	0.1	6.3	0.62
C, 20' - 30'	0.8	8.6	0.83	2.0	7.2	0.76	2.0	5.5	0.66	0.5	5.8	0.60
D, 30' - 40'	0.7	8.7	0.84	1.7	7.4	0.77	2.0	5.4	0.71	0.7	5.7	0.61
E, 40' - 50'	0.5	8.7	0.83	1.5	7.5	0.78	1.8	5.5	0.67	0.1	5.4	0.59
No. 3012 (Hole D2)												
A, 0' - 10'	0.7	8.9	0.85	1.7	7.7	0.81	2.7	5.4	0.70	1.8	5.0	0.68
B, 10' - 20'	0.5	8.7	0.82	1.5	7.6	0.78	2.0	5.7	0.70	0.6	6.0	0.62
C, 20' - 30'	0.7	8.6	0.83	1.7	7.4	0.79	2.0	5.7	0.70	1.0	5.6	0.65
D, 30' - 40'	0.7	9.0	0.84	1.3	7.8	0.80	2.0	6.0	0.71	1.0	5.8	0.65
E, 40' - 50'	0.7	8.4	0.85	1.7	6.8	0.78	2.0	5.2	0.69	1.8	4.7	0.62

Shr = Linear Firing Shrinkage, % of wet length, directly additive to drying shrinkage.

Abs = Absorption, 5-hr boil basis, % of dry, fired weight.

C/B = Saturation Coefficient.

3. There is less than one cone difference in PCE among the 10 samples.

4. Differences in fired shrinkage and absorption between the 10 samples are little more than expected from normal experimental error.

DTA indicates minor differences in clay mineralogy between the two holes. Hole D2 (Sample No. 3012) contains the more plastic minerals, as evidenced by consistently higher dry strengths. The greatest apparent differences, however, are probably the changes in hardness and plasticity with depth of hole. The plastograms show decreasing plasticity with depth, which is reflected in the increased forming pressures required for stiff extrusion. This factor is unlikely to be of major significance, inasmuch as additions of plastic clay are desirable to improve extrusion behaviour and to increase dry strength.

/MS